

# LABNOTES *Spring 2004*

## Top-Notch Laboratories Honored by the DNR

By Camille G. Johnson

On March 24, 2004 at the Natural Resources Board Meeting the 2004 Registered Laboratories of the Year were honored. DNR Secretary Scott Hassett and Environmental Science Services Section Chief David Webb presented the awards to Fontana-Walworth Water Pollution Control Commission Laboratory and Village of Boyd Wastewater Treatment Plant Laboratory.



*The Village of Boyd's Tom Grunewald (L) accepts his laboratory's award from DNR Secretary Scott Hassett and ESS Section Chief, David Webb (R).*



*Secretary Scott Hassett (2<sup>nd</sup> from right) presents Fontana-Walworth's Janet Tiffany and the rest of Fontana-Walworth's crew, with their award. ESS Section Chief David Webb is on the far right.*

This is the ninth year these awards have been presented to laboratories that have demonstrated exceptional efforts towards

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*Labs of the Year, continued.*

generating high quality data. The data they generate is very important because many programs in the DNR utilize data submitted by these labs to make regulatory decisions. Registered Laboratories may be nominated for this award by anyone, and a committee made up of DNR Lab Certification personnel chooses the winners. David Webb stated that "over 300 registered laboratories were eligible to be chosen for this honor". It takes very outstanding efforts to be nominated for this award, and to be chosen as the winner is a great mark of distinction.

The **Fontana-Walworth Water Pollution Control Commission Laboratory**, located in Walworth, was awarded the Laboratory of the Year Award in the Large Registered Facility category. They analyze wastewater samples for ammonia, phosphorus, biochemical oxygen demand (BOD), total suspended solids (TSS) and chloride for the municipal wastewater facility.

DNR Audit Chemist, John Condron, nominated the laboratory. In his nomination Mr. Condron wrote about the lab's "...painstaking attention to important detail with the corresponding documentation of those details." He also pointed to the fact that no deficiencies were identified at their last lab evaluation and they do more than the minimum quality control practices. Their very restrictive control limits and innovative lab practices also impressed Mr. Condron.

When presenting the award, David Webb said that the Fontana-Walworth Lab "...goes the extra mile and has a long history of excellent on-site evaluations". The facility was also honored for their mastery of the tests they perform, and strong measures taken to ensure the production of high quality data. When accepting the award, Janet Tiffany, the Fontana-Walworth Laboratory Director, thanked the whole Fontana-Walworth Commission, and the Bowman-Mealy training team for all their help.

*Continued on next page.*

## LabNotes

### Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

John R. Sullivan, Director  
Bureau of Integrated Science Services  
(608) 267-9753

David Webb, Chief  
Environmental Science Services Section  
(608) 266-0245

Rick Mealy  
LabNotes Editor  
(608) 267-7633

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*Lab of the Year winners, continued.*

The Village of Boyd Wastewater Treatment Plant Laboratory, located in Boyd, was awarded the Laboratory of the Year Award in the Small Registered Facility category. The Boyd Lab analyzes municipal wastewater samples for biochemical oxygen demand and total suspended solids.

DNR Environmental Engineer, Steve Thon and DNR Audit Chemist, Camille Johnson nominated the laboratory. In his nomination Steve Thon wrote, "[Boyd]...will stand as an excellent example of how a small lab, with staff that must perform a variety of unrelated tasks, operating with limited resources, can not only achieve the goal of high quality data, but also provide useful and timely information for plant operation". The other nominator, Camille Johnson, wrote that the main operator Tom Grunewald "has a strong work ethic and his efforts in the lab are consistently yielding excellent results".

**Tom Grunewald "has a strong work ethic and his efforts in the lab are consistently yielding excellent results"**

When presenting the award to Boyd, Mr. Webb stated that "there were no deficiencies cited at the last evaluation and preventative maintenance is a top priority". The facility was also honored for doing more quality control than required, having a very organized, albeit tiny, laboratory and for their superb results.

Nominations for the 2005 Registered Laboratory of the Year awards are now being accepted. Anyone may nominate a facility as long as they fall into the Registered category (labs in the Certified category are not eligible). To obtain a nomination form contact Camille Johnson at (715) 831-3272 or by email at [Camille.Johnson@dnr.state.wi.us](mailto:Camille.Johnson@dnr.state.wi.us). □



## Meetings & Training Opportunities

### Operator Certification Exams

DNR will hold Wastewater, Drinking Water and Septage Operator Certification exams November 3, 2004 (*postmark deadline October 6, 2004*) in DNR Regions around the state. Check the Operator Certification web site for details, as they become available. Application packets were mailed in February 2004.

[www.dnr.state.wi.us/org/es/science/opcert](http://www.dnr.state.wi.us/org/es/science/opcert) □

### 2004 Conferences, Meetings

#### WLA 28<sup>th</sup> Annual Conference

The Wisconsin Laboratory Association (WLA) will hold the 28<sup>th</sup> Annual Educational Conference, September 15 and 16 at Liberty Hall in Kimberly (near Appleton). The purpose of WLA is to provide laboratory staff in various fields including water, wastewater and hazardous waste with educational programs emphasizing technical information, new laboratory technologies and networking opportunities.

The WLA was founded by a group of concerned laboratory personnel in 1976 as a professional organization for persons involved in non-clinical laboratories in the state of Wisconsin. The goal of the organization is to promote the level of professionalism of all personnel involved in the analytical laboratory field through the Annual Educational Conference, workshops and scholarship program.

The WLA website will be updated with conference information including registration, as it becomes available.

*Continuing education credits are available for one or both conference days.*

<http://wilabassociation.tripod.com/wla/id1.html> □

## WWA Annual Conference

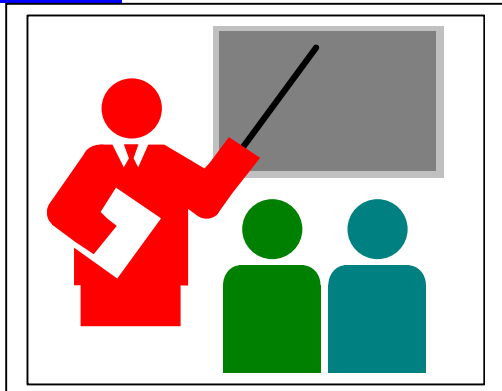
The Wisconsin Water Association (formerly AWWA WS) annual conference is scheduled for September 15 through 17, 2004 in Appleton. Contact Jack Albrechtson at (608) 831-6554 for more information.

[www.wiawwa.org](http://www.wiawwa.org) □

## WOWA Annual Conference

The Wisconsin Wastewater Operators Association annual conference is to be held October 26 through 29, 2004 at the Kalahari Resort in the Wisconsin Dells. Check the WOWA web site for more details.

[www.wwoa.org](http://www.wwoa.org) □



## Training for Lab Analysts

**Laboratory Analysis 1**      September 7-9, 2004  
Fond du Lac/MPTC      (800) 221-6430

**Laboratory Analysis 2**      November 9-10, 2004  
Green Bay/NWTC      800 422-6982 x5444

**WaterWorks Math**      November 10, 2004  
Madison/MATC      608-246-5217 (Barb) or  
608-246-5201 (Don)

**WaterWorks Math**      November 11, 2004  
Green Bay/NWTC      800 422-6982 x5444

BTC: Blackhawk Technical College

CVTC: Chippewa Valley Technical College

FVTC: Fox Valley Technical College

NWTC: Northeast Wisconsin Technical College

MPTC: Moraine Park Technical College

MATC: Madison Area Technical College

[www.dnr.state.wi.us/org/es/science/opcert/training.pdf](http://www.dnr.state.wi.us/org/es/science/opcert/training.pdf) □

## Program Administration

### New Look for Web Site



[www.dnr.state.wi.us/org/es/science/lc/](http://www.dnr.state.wi.us/org/es/science/lc/)

### Laboratory Certification

### Program Information

### Rules

### Application Process

Application Forms

Current Application Fees

Application Fees (After July 1)

Special Application Requirements

Application Timeline

Application Expiration

Certification Renewal

On-Site Evaluations

In early March, the DNR server went "live" with a complete overhaul of the Laboratory Certification and Registration Program's website. On most pages now you will find side button links and sub-links to particular aspects of the Program. Major buttons have been created to reflect the six main aspects of the Program, some of which were only indirectly referenced previously.

Designed to address frequent questions asked by phone callers, our hope is that the wealth of information available on our site is more readily accessible to the public.

Many of the changes to the Lab Cert. web pages are technical and occur behind the scenes. But a few changes have resulted in information not being in the same place web users were used to finding it.

Please take the opportunity to check out our redesigned web pages—your comments will help us make sure we are meeting your information needs. □

## ***Fiscal Year 2005 Certification and Registration Fees***

The Natural Resources Board on March 24 unanimously approved the department's laboratory certification and registration fee schedule for Fiscal Year 2005. The fee schedule was previously reviewed by the Certification Standards Review Council, who provided their unanimous support and recommended Board approval. A public meeting was held on February 16 in DeForest, WI to discuss the proposed fee schedule. There were no attendees. The approved fee schedule will allow the Department to fund the laboratory certification and registration program at a level \$47,000 below its spending authority as established under Chapter 20.370(3)(fj), Wis. Stats.

The complete fee schedule is provided in the table below:

**Laboratory Fees for FY 2005 (Sept.1, 2004 - Aug. 31, 2005)**

<b>Fee Item</b>	<b>FY 2005 Unit Price</b>	<b>Fee Item</b>	<b>FY 2005 Unit Price</b>
Registered Base Fee	\$540.00	Category 10	\$216.00
Certified Base Fee	\$810.00	Category 11	\$216.00
Reciprocity Fee	\$1,620.00	Category 12	\$216.00
<b>Initial Application Fee</b>	<b>\$324.00</b>	Category 13	\$216.00
<b>Revised Application Fee</b>	<b>\$162.00</b>	Category 14	\$216.00
Category 1	\$54.00	Category 15	\$648.00
Category 2	\$54.00	Category 16	\$216.00
Category 3	\$54.00	Category 17	\$648.00
Category 4	\$54.00	Category 18	\$1080.00
Category 5	\$108.00	Category 18a (Nitrate Only)	\$108.00
Category 6	\$108.00	Category 18b (Nitrate & Fluoride)	\$216.00
Category 7	\$216.00	Category 19	\$216.00
Category 8	\$216.00	Category 20	\$1,404.00
Category 9	\$216.00	Category 21	\$216.00

***Note: Application fees are effective July 1, 2004***

Fees are calculated using the formula promulgated in s. NR 149.05, Wis. Admin. Code. This formula uses a relative value system to equitably distribute the cost of administering the program across all participating laboratories. Each fee item is assigned a relative value in Ch. NR 149, Table 2. The total number of available RVUs is the sum of the relative values of each fee item multiplied by the number of labs certified or registered for that fee item in the coming fiscal year. The cost per RVU is calculated by dividing the program's operating costs (not including projected travel costs for audits of out-of-state labs, for which these labs are billed directly), by the total number of available RVUs. The cost of each fee item is determined by multiplying its relative value by the cost per RVU.

Certification and registration renewal fees will appear on the environmental fee statements that will be mailed in late May. Payment will be due in full by June 30, 2004. Late fees will be assessed to laboratories that fail to pay renewal fees by this deadline.

Please contact Greg Pils at (608) 267-9564 or gregory.pils@dnr.state.wi.us if you have any questions about your fees. □



### **Alternative Bottles and Filters for BOD and TSS**

Over the past few months, we've been asked if it's acceptable when analyzing samples for TSS to use filters other than those specifically referenced in Standard Methods 2450 D and EPA 160.2. The answer is "yes", provided that they are glass fiber filters, do not contain organic binder, and give "demonstrably equivalent results" – which means that they should give you results that compare favorably with those listed in the methods. It's important to note also that these filters do not require approval by EPA or any other authority.

#### **Answering the questions:**

- ◆ Are the new "non-glass" BOD bottles acceptable?
- ◆ Can TSS filters other than those listed in Standard Methods be used?

Unlike TSS filters, BOD bottles constructed from materials other than glass do require EPA approval before they can be used, as the 5-Day BOD method specifically requires the use of glass bottles. If you're interested in using bottles constructed from plastic or other non-glass material, check with the vendor to see if their product has received EPA approval, and ask for a copy of the EPA approval letter for your records. Auditors will ask for such approval verification while on-site, and laboratories will be cited as deficient if they can not produce verification that EPA has approved the non-glass BOD bottle. These approvals are granted for each vendor's specific product. One vendor's approval does not mean that another vendor's product is approved simply because the construction material is the same.

Please contact Greg Pils at (608) 267-9564 or [gregory.pils@dnr.state.wi.us](mailto:gregory.pils@dnr.state.wi.us) if you have any further questions about BOD bottles and TSS filters.

### **SDWA Certification Requires PT by Method**

Safe drinking water act certified laboratories are required to annually achieve acceptable results on PT samples for each analyte/analyte group and **for each method used to report compliance monitoring results**. Methods used solely for confirmation are excluded. To be certified for an analyte group (e.g., VOCs, haloacetic acids) laboratories must pass 80% of the individual analytes in the PT sample.

The requirement to analyze PT samples by each method used is located in the EPA's "Manual for the Certification of Laboratories Analyzing Drinking Water," 4<sup>th</sup> ed. March 1997. The Wisconsin Laboratory Certification and Registration Program rule incorporates the EPA Drinking Water Certification Manual by reference (see s. NR 149.21, Wis. Adm. Code). On December 1, 1999, EPA promulgated the requirement for PTs by method in the Federal Register, with an effective date of January 1, 2000. In Wisconsin, implementing the requirement for PTs by method is complicated by the fact that certification is offered by analyte and not by method. However, this does not exempt laboratories from meeting the federally promulgated requirement, since Wisconsin, as a primacy state, holds delegated authority. The Wisconsin Laboratory Certification and Registration Program requires laboratories submitting applications for SDWA analytes to include PTs, MDL studies, and --for organic analytes--IDC studies, for each method listed on the application.

The Laboratory Certification Program will be sending out a status update form to each laboratory currently certified or registered to perform drinking water analyses. Laboratories must report back on these forms the approved methods that they intend to use to analyze drinking water compliance samples. In order to continue to provide compliance data to Wisconsin, laboratories will be required to pass a WS sample from an approved provider for each analyte/analyte group and using each method indicated on the form. Compliance results submitted for any parameter for which a laboratory has not submitted the requisite PT information will not be accepted by the Department.

Please contact Rick Mealy at (608) 264-6006 or [richard.mealy@dnr.state.wi.us](mailto:richard.mealy@dnr.state.wi.us) if you have any further questions this requirement.

## Renewal Reminders

Last September a substantial number of laboratories in the Program found themselves with gaps in their certifications due to problems during the renewal process. Our program does not have a formal, annual re-application process. At the risk of over-simplifying things, all that is required to renew your certification is:

- (1) pay your annual fees prior to August 31
- (2) analyze, pass, and forward to us a PT sample --from an approved provider-- for each analyte or analyte group for which you hold certification (and a PT is required).
- (3) If your laboratory holds reciprocal certification with Wisconsin, please be sure to send us an updated certificate from the originating state. Please also make sure to send us a copy of your latest originating state's audit report .

The single greatest cause for non-renewal of tests or test categories is related to missing PT samples. In addition to the fiscal (application fees) burden, failing to provide these things can result in loss of valuable clients or hamper site investigations. Department Programs may not accept data generated during a lapse in certification. While the Laboratory Certification and Registration Program sends letters out each June to those facilities that are lacking acceptable PT sample results for one or more parameters, there are some things each lab should do to ensure that certification renewal progresses smoothly.

Register with an approved provider for each certified parameter as soon as possible after January 1 each calendar year. PT sample results issued prior to January 1 of each calendar year cannot be used for annual certification renewal, which begins each May. A list of certified PT providers' contact info is available at: [www.dnr.state.wi.us/org/es/science/lc/PT/PT%20Provider%20Contact%20Info.pdf](http://www.dnr.state.wi.us/org/es/science/lc/PT/PT%20Provider%20Contact%20Info.pdf)

A list of parameters that each certified provider has been approved to provide for Wisconsin certification is available at: [www.dnr.state.wi.us/org/es/science/lc/PT/PT%20Provider%20Parameter%20Approvals.pdf](http://www.dnr.state.wi.us/org/es/science/lc/PT/PT%20Provider%20Parameter%20Approvals.pdf)

Participating in a study early will provide you with sufficient time to participate in a remedial study for any parameters scored as unacceptable.

Direct renewal-related questions to Rick Mealy at (608) 264-6006 or via e-mail to

[richard.mealy@dnr.state.wi](mailto:richard.mealy@dnr.state.wi).

## Clarifications from Fall '03 LabNotes

### Wastewater Preservation for Ammonia

Camille Johnson

This is a clarification to the previous article in Fall 2003 Labnotes titled "Ammonia Testing in Wastewater". The article was geared towards ammonia samples collected from wastewater treatment plants and sent to a certified lab. The preservation techniques were unclear to some of our readers. This was partly due to the fact that Standard Methods has different guidelines for ammonia preservation, but NR 219 is the Wisconsin code that must be followed. NR 219 requires preservation of ammonia by cooling to 4 °C, acidifying to pH 2 or below with concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and holding the sample for a maximum of 28 days. If the sample is going to be analyzed for ammonia on-site at your own laboratory that is registered or certified for ammonia analysis, then the acid does not have to be added if the samples are analyzed immediately (within 15 minutes). If you cannot run the analysis until a later time then the preservation techniques must be followed.

### Clarification of LOD & Reporting Requirements

Rick Mealy

In the last edition of LabNotes (January 2004), we provided a tabular listing of those wastewater parameters for which the LOD and LOQ must be reported on monthly DMR forms. Some facilities have apparently mis-interpreted the extent of this information. DMR requirements are independent of Laboratory Certification requirements. This means that while you may not be required to report LOD and LOQ for a specific analyte on your facility's DMR form, it does not exempt the facility from a basic Laboratory Certification requirement: that each facility must establish--and verify annually-- an LOD for each certifiable parameter.

----- LOD/LOQ -----		
Parameter	Must have?	Report On DMR?
T.res. Chlorine	YES	YES
NH3-N	YES	YES
-----		
TP	YES	no
TSS	YES	no
BOD5	YES	no
cBOD5	YES	no

**State of Wisconsin \ DEPARTMENT OF NATURAL RESOURCES**

Jim Doyle, Governor  
Scott Hassett, Secretary

101 S. Webster St.  
Box 7921  
Madison, Wisconsin 53707-7921  
Telephone 608-266-2621  
FAX 608-267-3579  
TTY Access via relay - 711

April 22, 2004

To All Permittees Receiving the DMR-QA #24 Mailing

Dear Permittee:

You will soon be receiving or may have already received a packet of information for participating in EPA DMR-QA Study 24. On February 9, 2004, the Wisconsin Department of Natural Resources informed the Environmental Protection Agency (EPA) that Wisconsin permittees **will not** participate in this year's study or future years' studies. You are **not required** to perform steps called for by the Study instructions to remain in good standing with your Wisconsin Pollutant Discharge Elimination System (WPDES) permit.

Each laboratory that performs WPDES permit-required testing for Wisconsin permittees must hold a valid certification or registration under chapter NR 149, Wisconsin Administrative Code for each analyte for which it performs testing. Under chapter NR 149, the Laboratory Certification Program requires that laboratories meet performance criteria on proficiency samples by September 30 each year to maintain certification or registration status. Laboratories may use proficiency samples from any of the currently approved providers. If you meet the requirements for Wisconsin Laboratory Certification proficiency testing, there is no need for you to participate in the EPA DMR-QA Study.

Over the last few years, DNR staff have explained to EPA that in-place management systems in Wisconsin, including our Laboratory Certification Program, makes the DMR-QA studies unnecessary for insuring the quality of Discharge Monitoring Report data. Because the Department does not directly control the distribution of DMR-QA study materials, we were not able to prevent this year's package from being sent to you. We hope this letter reaches you in time to minimize your effort in response to that mailing.

The Wisconsin Department of Natural Resources is committed to using the best quality data possible for evaluating efforts in protecting water quality. However, we cannot afford activities that result in an unnecessary duplication of resources.

Should you have questions, please contact Tom Mугan at 608 266-7420 ([tom.mugan@dnr.state.wi.us](mailto:tom.mugan@dnr.state.wi.us)) in the Bureau of Watershed Management or Rick Mealy of the Laboratory Certification Program at 608 264-6006 ([mealyr@dnr.state.wi.us](mailto:mealyr@dnr.state.wi.us)).

Sincerely,

Russ A. Rassmussen, Director  
Bureau of Watershed Management

John R. Sullivan, Director  
Bureau of Integrated Science Services

**RECEIVED**

cc Dr. Clyde Marion - WC-15J, EPA Region V  
Dr. John Helm - (2225A), EPA Office of Compliance

APR 26 2004

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## Proficiency Testing

***Wisconsin will no longer require DMRQA samples***



Historically, DMRQA samples provided to permittees were simply extra ampules from the previous year's WP study. As far back as 1997, the Department was successful in removing permitted facilities from the DMRQA mailing list due to involvement in other equivalent PT programs. Years ago, a number of facilities relied on DMRQA PT results to satisfy their annual certification requirements for the Laboratory Certification and Registration Program. Schedules have since changed, however, and labs no longer have sufficient time to receive their DMRQA results before renewal deadlines, particularly if unacceptable results are obtained for one or more parameters. These realities have forced laboratories to participate in other PT studies to satisfy their annual certification requirements. As has been the case historically, the WP PT studies laboratories now participate in are in every way equivalent to DMRQA studies.

Beginning with this year (DMRQA Study #24), The Department of Natural Resources' Watershed Management Program will no longer require analysis of annual DMRQA proficiency testing samples for compliance with WPDES permit requirements. Working with the Laboratory Certification & Registration Program, Watershed Management staff determined that the DMRQA PT samples represent a redundancy of PT requirements necessary to maintain Wisconsin laboratory certification. In the interest of always trying to find efficiencies and streamline processes, we cannot afford to administer a redundant program that offers no added value. A copy of the letter sent to all permitted facilities appears on the adjoining page.

The Department of Natural Resources and the Laboratory Certification Program take data quality very seriously. We realize that the purpose of the DMRQA studies is to ensure that high quality data is generated in implementing the Clean Water Act. We are confident that the Laboratory Certification Program's PT requirements satisfy the objectives of the DMRQA studies. In addition, we believe the strength of our on-site evaluation program, in conjunction with on-site evaluations performed by basin engineers, provides us with an excellent early warning system to identify and correct deficient practices before data quality is affected.

### ***Deadlines for Renewal PTs***

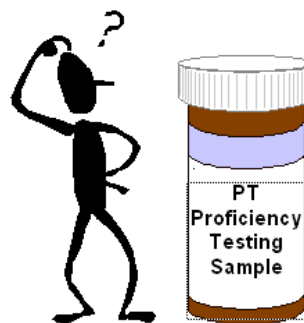
**January 1** PT studies must close after January 1 to be counted for the 2004-2005 certification and registration cycle.

**August 31** Acceptable results must be received by the Department by midnight.

**September 1** Laboratories that did not submit acceptable reference sample results for each test for which they are required prior to September 1 are not renewed for those tests, must cease performing analyses for the analytes, and are required to subcontract the work to a certified laboratory. Reapplication is necessary.

Laboratories must annually achieve acceptable reference sample results for each test for which certification or registration renewal is sought. Reference samples for renewal must be analyzed after January 1 of each calendar year. This office must receive reports from reference sample providers by August 31. For example, if your laboratory wishes to renew its BOD certification for the period beginning September 1, 2004, you would have to analyze and pass a reference sample between January 1 and August 31, 2004. Although the current certification period ends August 31, 2004, the program needs sufficient time to generate and distribute certificates to the laboratory community by September 1.

Please direct questions about reference sample requirements to Rick Mealy, Laboratory Certification Chemist at (608) 264-6006 or [richard.mealy@dnr.state.wi.us](mailto:richard.mealy@dnr.state.wi.us).



## State Lab of Hygiene moves up its 3<sup>rd</sup> round of testing

The third shipment of the Environmental Reference program has been moved from September to June 15, 2004. This will enable laboratories who experience unsuccessful performance in the April event to obtain samples from WSLH PT and successfully analyze them before the Wisconsin DNR Laboratory Certification Program deadline of August 31<sup>st</sup>.

If your lab does not pass all analytes in the April event, you will automatically receive and be billed for the analytes that still need to be passed unless a written cancellation request is received by WSLH PT.

If you have any questions, please contact Barb Burmeister, Environmental PT Coordinator, at (800) 462-5261, ext. 107.



## Council Corner

By Paul Junio, Council Chair



The Commercial Laboratories constituency is probably the easiest constituency to represent on the Lab Certification Council. The constituency is well-defined, has an organization that meets on a quarterly basis, and is always ready to provide feedback, ideas, and criticism that can be brought forward to the Council. This doesn't mean that all commercial labs are properly represented, since not all labs are members of the Wisconsin Environmental Laboratories Association ([www.wislabs.org](http://www.wislabs.org)). If you fall in that category, please feel free to contact me!

If you're reading this, you have a Representative, guaranteed. Whether it's the catch-all of "Demonstrated Interest" or a defined segment of the laboratory testing business, there's someone named to the Council with whom you can interact.

Get involved! Is there something that you don't like about the Certification Program? Is there a change that you'd like to see made? How about a suggestion to make something better. Maybe you've run into a requirement made by some other Agency that relates to laboratory analysis. If so, please bring that to the attention of your Council Representative.



I was talking with a former Council member recently (Hi, Dave!), and he commented on where the program has advanced over the last few years. It's encouraging to see the limited number of labs that have not been audited in the past 4 years (or longer). Keep up the good work!



With the good comes the bad. While not an indictment of the Lab Certification Program, there have been some recent happenings where other sections of the DNR have put forth lab requirements without consulting Laboratory Certification. If this hits home with you, consider the effect of any requirements that may be written of laboratories that DON'T appear in NR149, and make sure that they are reviewed by the appropriate people before they become effective.

Current Council Members		
Representation	Name	Phone/ e-mail
Commercial Laboratory	Paul Junio (Chair)	(920) 261-1660 PJunio@testamericainc.com
State Laboratory of Hygiene	George Bowman (Vice-Chair)	(608) 224-6279 gtb@mail.slh.wisc.edu
Demonstrated Interest in Lab Certification	Marcia Kuehl (Secretary)	(920) 469-9113 Makuehl@aol.com
Public Water Utility	Katie Edgington	(608) 755-3115 edgingtonk@ci.janesville.wi.us
Small Municipal Wastewater Plant	Randy Herwig	(608) 592-3247 rherwig@wppisys.org
Industrial Laboratory	Jim Kinscher	(262) 636-1278 j.t.kinscher@na.modine.com
Large Municipal Wastewater Plant	Kurt Knuth	(608) 222-1201 x293 kurtk@madsewer.org

# Hazardous Waste

## ***Updates to Hazardous Waste (NR 600 series), Used Oil (NR 590) and PCB (NR 157) rules***

**By John Melby, Policy Chief**

One of the major Bureau of Waste Management work efforts for the next year or so is to update our Hazardous Waste (NR 600 series), Used Oil (NR 590) and PCB (NR 157) rules. Our hope is to have new rules approved by our Natural Resources Board (NRB) in October 2005.

Early in the spring of 2002, the Bureau of Waste Management started the process of revising our NR 600 series and NR 590 rules. Our goal is to update these rules and our authorization to administer the hazardous waste program, by paralleling the Federal rules as much as possible. The reasons for this change are as follows:

- The existing Hazardous Waste (NR 600 series) and Used Oil (NR 590) rules are outdated,
- More federal rules have been developed and we need additional EPA authorization for these,
- Many errors have crept into the rules over the years,
- Many Wisconsin-unique provisions add complexity and may not be needed to effectively implement the hazardous waste program,
- Keeping the rules consistent with EPA using the Wisconsin-unique rules organization is difficult and often confuses the public on what is required, and
- The DNR Waste Management Program must develop Wisconsin-unique guidance rather than using EPA guidance, which may not be an effective use of limited resources.



Department staff have translated the Federal rules into the Wisconsin rule format, compared them to our existing NR 600 series and NR 590 and drafted new rules to replace our current series. In the rule development process, staff are identifying the unique provisions that are in the existing NR 600 series and NR 590 rules and making recommendations to only keep the Wisconsin unique provisions when required:

- By Wisconsin statute,
- To addressing documented public health or environmental problems,
- To allow effective operation of the hazardous waste program,
- To maintaining consistent facility standards with other appropriate WI environmental programs, and
- To encourage safe recycling, and
- Current source of program revenue.

Here is the timing of our process:

- Natural Resources Board (NRB) approval to go to public hearing in October 2004,
- Proposed rules will be placed on the DNR Waste Management Program / Hazardous Waste website by November 1, 2004,
- Public hearings at several locations will occur in December 2004 and January 2005,
- Public hearing comments will be reviewed, a responsiveness summary developed and posted on the DNR Waste Management Program / Hazardous Waste website by July 15, 2005, and
- Natural Resources Board (NRB) final approval of the rules in October, 2005.

Requirements to have Wisconsin rules that are as stringent as the Federal rules, upon which they are based, limit our ability to relax rules. However, we will do our best to provide opportunities to comment on the proposed rules and to have a rule development process that is as transparent as possible. Thanks for your interest and more to come as the rule development process unfolds.

## Drinking Water

### *Electronic Reporting Update*

By now, you should be aware that the Wisconsin DNR will require that all public drinking water compliance data be submitted to the the Department electronically. The target date for this requirement is January 1, 2005. Many laboratories responded to a survey that was sent to all SDWA-certified labs and many have already begun planning their move to electronic reporting.

DNR mailed out 127 surveys last January and received 78 in response. Of those that responded, all but one indicated that they would be able to use one of the three electronic reporting formats that DNR has proposed. 55% indicated that they would prefer to use the internet to enter data on-line. 29% reported an interest in using a tab-delimited text file format and 11% preferred to use XML. The web-based form is still being developed by DNR and should be available by early fall. Labs that plan to utilize one of the approved file transfer formats (tab-delimited or XML) can begin their own development at any time.

One interesting note is that even though the majority of labs are interested in a web-based data entry format, they don't account for the majority of samples analyzed in the state. Most larger-volume labs will likely use their existing information management systems to extract data and transfer files to DNR.

The Department will go to the Natural Resources Board in May with a request to hold public hearings on the proposed changes to ch. NR 809 that will require electronic reporting. The hearings would be conducted this summer, with locations yet to be determined. We also plan to hold several workshops this fall to assist laboratories with the transition.

Information on the electronic data process, with example file formats can be found at:  
[www.dnr.state.wi.us/org/es/science/lsl/lab\\_data/file\\_layout.htm](http://www.dnr.state.wi.us/org/es/science/lsl/lab_data/file_layout.htm)

For more information, you may contact either

Gail North [Gail.North@dnr.state.wi.us](mailto:Gail.North@dnr.state.wi.us)

(608) 264-6131 or

Ron Arneson at [Ronald.Arneseon@dnr.state.wi.us](mailto:Ronald.Arneseon@dnr.state.wi.us)

(608) 264-8949

## Cross Media Issues

### *Arsenic Standards are being Lowered*

State Administration Code, chapter NR 140, for groundwater quality standards has been revised for arsenic (As). The new standard was effective on March 1, 2004. These standards were changed to be consistent with the current federal standard for drinking water. Comparisons of the old and new standards are given below.

	Previous	New
Groundwater Preventive Action Limit (PAL)	5 ug/L	<b>1 ug/L</b>
Groundwater Enforcement Standard (ES)	50 ug/L	<b>10 ug/L</b>
Drinking Water Maximum Contaminant Limit (MCL)	50 ug/L	<b>10 ug/L</b>

A "PAL" (preventive action limit) is a numerical value expressing the concentration of a substance in groundwater which is adopted under s. 160.15, Stats., and s. NR 140.10, 140.12 or 140.20.

An "ES" (enforcement standard) is a numerical value expressing the concentration of a substance in groundwater which is adopted under s. 160.07, Stats., and s. NR 140.10 or s. 160.09, Stats., and s. NR 140.12.

"MCL" (maximum contaminant level) is the maximum permissible level of a contaminant in water which is delivered to any user of a public water system.

The administrative code can be found on the Revisor of Statutes web site at the follow address:  
<http://www.legis.state.wi.us/rsb/code/nr/nr100.html>

The state drinking water standard (MCL) is also being revised to be consistent with the federal standard.

You should evaluate your ability to quantify groundwater samples at these levels. Inductively coupled plasma-atomic emission spectrometry (ICP-AES) may not be able to detect and quantify at these levels. This would include EPA methods 6010B and 200.7.

# Wastewater Forum

## BOD Hints - Seed

If you use synthetic seed (i.e. Bioseed ©) to seed your BOD/GGA tests be sure to mix the seed properly. The seed must be mixed with the correct amount of DILUTION water, not plain distilled or other water types. The quantity of dilution water needed should be specified on the commercial seed bottle. The seed will not yield healthy, viable population of microorganisms ("bugs") if you use distilled water because the ionic balance of the water is so poor that much of the seed will not survive.

## BOD Hints - GGA

When making your GGA standard it is very important to have the standard at room temperature. The best way to do this is to pour out about 7-9 mLs of the GGA standard into a small beaker and allow it to come to room temperature (this could also be carefully sped up by placing the beaker in a warm water bath). Once the standard is at room temperature then 6 mL of the standard should be measured with an accurate pipet. If you measure your standard while it is still cold you will end up putting more than 6 mL of the standard into your BOD bottle which can in turn cause you're your GGA standards to be biased high.

## Water Extractable Phosphorus (WEP) in Biosolids

The department is assessing the relative environmentally available phosphorus between biosolids, manure, and commercial fertilizer. Early indications show that the water extractable phosphorus in most biosolids is much less than that found in manure or commercial fertilizer. Further the iron, aluminum, and oxides that are commonly found in biosolids serve to form strong and long-lasting bonds with the phosphorus. To gather more relevant information, all municipal biosolids and industrial sludge producers are requested to begin testing for water extractable phosphorus (WEP) in addition to the total phosphorus testing already required in permits. The recommended test

method as developed by researchers at Penn State University is called "Water Extractable Phosphorus", or WEP.

The WEP procedure involves the extraction of an amount of biosolids equal to 0.5 grams dry weight to which a volume of deionized/distilled water is added to make the total weight of sample plus deionized/distilled water equal 100.5 grams. The sample is then placed on a shaker table, providing moderate shaking, for one hour, followed by direct analysis (no digestion) by either ICP or the single reagent, ascorbic acid, colorimetric (i.e., Murphy-Riley) procedure. The procedure for reporting results is summarized below:

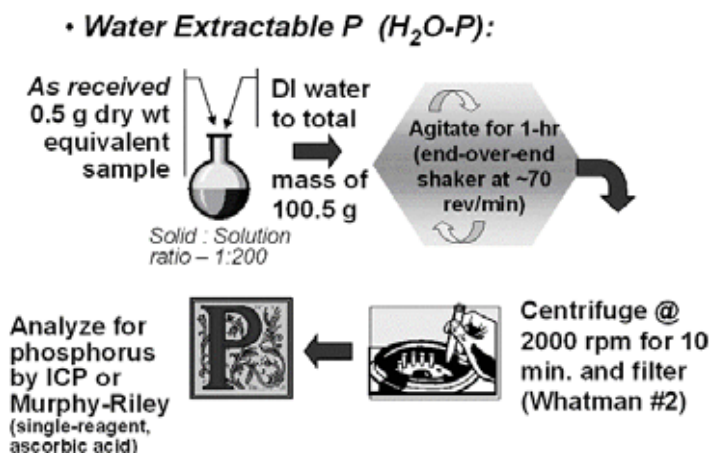
1. Analyze **Total P** (*digested*)
2. Extract and Analyze **WEP** (*no digestion*)
3. WEP result =  $\frac{\text{WEP mg/kg dry wt}}{\text{TP mg/kg dry wt}} \times 100$

The complete procedure, developed at Penn State University, can be obtained from our website at:

<http://www.dnr.state.wi.us/org/es/science/lc/OUTREACH-Methods/WaterExtractablePhosphorus.pdf>

The complete letter that was sent out to permittees can be viewed on our website at:

[http://www.dnr.state.wi.us/org/es/science/lc/OUTREACH-Guidance/151\\_07ImplementationMemo.pdf](http://www.dnr.state.wi.us/org/es/science/lc/OUTREACH-Guidance/151_07ImplementationMemo.pdf)





# General Interest Articles

## ***R UR ICP IECs A-OK?***

By Rick Mealy

One thing I've noticed over the course of more than 10 years of auditing laboratories is that, as in the old party game, called--among other names,--"Telephone", many of the details of ICP analysis have been diluted out-- so to speak-- over the course of years of passing information down from analyst to analyst. In particular, I find that only infrequently do I encounter a laboratory that has adequately evaluated and verified interference correction in a technically sound manner. The focus of this article is to re-visit the early days of ICP and re-capture the critical aspects verification of interference identification, correction, and correction.

### **What's the purpose of an ICS?**

Once a lab has established its interference correction factors (how this is actually accomplished is a topic for a separate article), the goal of any evaluation solution, regardless of terminology employed, is to provide documentation that the inter-element correction (IEC) factors provide accurate correction over the range of routine samples analyzed. That last part is often overlooked, yet critical to proper correction. If IEC factors are either (A) not established or (B) not evaluated at interferent concentrations typically encountered, one may obtain acceptable results on the evaluation standards, or interference check standards (ICS), but actual sample results will be subject to bias.

Consequently, the goal of our ICS solutions is to provide a relatively challenging mix of interfering elements, without incorporating elements that exhibit spectral overlap with each other. To ensure that the samples pose a challenge, the interfering elements (both primary and secondary) that are routinely identified in samples should be included. In addition, the concentration levels to be tested should represent at least 95% of the maximum concentration that is typically encountered for each interferent.

How does this relate to what can be expected during an audit? Basically laboratories will be asked to demonstrate that they have adequately addressed the following:

- Has the laboratory properly identified

interferences due to spectral overlap

- Have interference correction factors been established? If not, is there adequate documentation that no interferences exist?
- Has the laboratory instituted an analytical QC program to verify that interferences have been appropriately identified and corrected?
- Does the laboratory's ICS have the ability to identify false positives related to spectral interference?

### **How should ICS solutions be prepared?**

Most labs follow the ancient EPA Contract Laboratory Program (CLP) approach of using an ICS-A solution (interferents only) plus an ICS-AB solution (interferents plus analytes of interest). In fact, vendors of mixed standards commonly sell these solutions prepared to match the concentrations specified in CLP Statements of Work (SOW). Interestingly, this approach was established in the early to mid 1980's, when a number of the available ICP instruments were not capable of displaying negative values. Early CLP protocol required the analysis of an ICS-A solution containing the major interfering cations (Al, Ca, Fe, Mg) at 500 ppm (Fe at 200 ppm). A second, ICS-AB solution was also required. This solution consisted of ICS-A to which 0.5 to 1.0 mg/L of each analyte of interest was spiked. These are typically the levels in solutions that laboratories can currently purchase from many vendors. Notable, however is that current versions of 200.7 (7.13.6) and 6010 (4.5) now specify that ICS solutions need only be spiked with elements of interest if the instrument is incapable of displaying negative values. Nearly every ICP purchased in the past 10 years is capable of displaying negative numbers.

Perhaps more important than analyzing target elements is to analyze ICS samples which contain the typical interferent elements. This will demonstrate that interferences stemming from these elements have been properly corrected. Initially, method 200.7 (17 elements) and 6010 (10 elements) direct the user to evaluate the elements listed in Table 2 for potential interferences. This seems like a reasonable place to start. Certainly, any element for which an IEC factor is established should be included in the ICS solutions.

The CLP's ICS-A solution actually seems like a very reasonable choice for an ICS. This solution contains the four major cations, two of which (Al, Fe) are elements that interfere with several other trace elements. Since many labs are already accustomed to analyzing a second solution, a second solution should not represent any analytical burden. A second

*Continued on next page.*



solution allows the lab to provide a measure of the effectiveness of IEC factors for secondary interferences.

Table 2 of EPA method 200.7 contains a number of analytes (e.g., cerium, molybdenum, titanium) that would only be expected to be encountered in unique samples. In most cases, there is little value in including such analytes in a second ICS solution. However, labs that routinely analyze industrial waste or biosolids may want to include Mo, as it is frequently detected in these samples. On the other hand, Table 2 does list several commonly found analytes (e.g., nickel, beryllium, cadmium, chromium) that should definitely be considered for inclusion in a second ICS solution, perhaps labeled ICS-B. Each laboratory should prepare an ICS-B solution that best fits the samples it typically encounters, and the analytical lines (wavelengths) it uses for analysis. Whatever selection is made, none of the elements included in any particular ICS solution should have documented interferences with any other element in that solution.

#### **How should ICS solutions be evaluated?**

Auditing a lab's ICP capability is one of the things I find most interesting in any laboratory. Although they represent the heart of ICP analysis, sections in the published methods (200.7 & 6010) related to interference identification, correction and evaluation are the most difficult to comprehend of any analytical method I've ever encountered. I'll typically ask the laboratory analysts if they've read the aforementioned sections of the method (always answered in the affirmative) and then follow up that question by asking if they understand what the method is specifying. Invariably, the answer is a firm negative. I'll admit that as many times as I've tried to go through these sections myself, I've had a difficult time following the direction provided.

In the absence of clear requirements or even guidance, the words that I have come to dread when I inquire how a particular laboratory evaluates its ICS solutions are: "plus or minus 20%". I realize, of course, that this answer has its roots in the CLP program, and can still be found within method 6010. That being said, however, criteria of " $\pm 20\%$ " [of true value] simply do not represent current technology, do not reflect "good science", and mask significant bias on trace level results. If one considers that ongoing calibration check standards are required to fall within 10% of true value for all elements and that ICS solutions are really no

different than standards, then the  $\pm 10\%$  criteria applicable to standards seem more realistic for ICS solutions. Criteria of  $\pm 20\%$  appear even more unrealistic when applied to ICS-AB solutions in which target analytes are present at about 1.0 ppm. This translates to an acceptable deviation of + 200 ppb, when LODs for these analytes are frequently in the 1-10 ppb range. A laboratory could be experiencing spectral overlap for one or more analytes that results in an apparent (false positive) concentration of as much as 20-100 times the LOD that would escape detection with such broad criteria. In fact, if a laboratory chooses to utilize the conventional ICS-AB solution to evaluate interference correction, it would be more appropriate to spike target analytes closer to the LOQ, rather than 0.5 to 1.0 mg/L, which represents at least 100 times the LOD of most analytes.

So...how should ICS samples be evaluated? To re-cap, solutions containing just the interferences should fall within  $\pm 10\%$  of the true value of each interferent. What's more important, and has not been discussed, is the evaluation of data from analytes that are not contained in the ICS solutions. Technically speaking, no one should argue that the analysis of any ICS solution that contains no target analytes should yield results of  $\pm$  the LOD for each target analyte. This is a defensible approach that ensures adequate identification and correction of any spectral overlap type interferences. If results for any particular (unspiked) target analyte are significantly negative (much below -LOD), then over-correction for an interference may be the culprit and requires investigation. Alternatively, a background correction point may occur in a region overlapped by the interfering analyte peak. Conversely, if an (unspiked) target analyte is detected to be present at a concentration greater than the LOD, insufficient correction of an interference or a poorly selected background point should be considered and investigated. Of course, this requires a laboratory to have established good, defensible LODs.

#### **How often should ICS solutions be analyzed?**

The CLP program historically required ICS-A and ICS-AB solutions to be analyzed at the beginning and end of each analytical sequence. This same frequency was adopted in earlier versions of SW-846 method 6010. Subsequent versions, however, require these solutions to be analyzed only initially with each analytical sequence. An allowance is provided to reduce the analytical frequency when a number of consecutive analyses indicate that IEC factors are working properly. □



## **LabNotes – Spring 2004, Volume 19, Issue 1**

Wisconsin Department of Natural Resources

101 South Webster Street

P.O. Box 7921

Madison, WI 53707-7921

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